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Structural and spectroscopic study of Fe-doped TiO_2 nanoparticles prepared by sol–gel method

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Abstract 5% (molar ratio) Fe doped TiO_2 nanoparticles were prepared by a sol gel method and the post annealing of the samples was carried out at 400 °C, 600 °C and 800 °C in air. Structural characterization of the samples was carried out using High Resolution Transmission Electron Microscopy (HRTEM). HRTEM images of the samples revealed that the mean size of the nanoparticles changed from ∼8 nm to ∼100 nm as the annealing temperature was increased. Experimental investigation of the electronic structure of TiO_2:Fe nanoparticles is important in order to understand the correlation between electronic and optical properties in these samples. X-ray Photoemission Spectroscopy (XPS) of the TiO_2:Fe nanoparticles was performed to study the electronic structure. The results of XPS study confirmed the presence of Fe in all samples, which could not be detected by HRTEM and XRD, in spite of low doping levels, and revealed that Fe ions are predominantly in Fe^{3+} states. Photoluminescence (PL) measurements have been measured with an excitation energy of 250 nm to study optical properties of the TiO_2:Fe nanoparticles. The active PL band at ∼440 nm has been observed.

1. Introduction

Nanocrystalline titanium dioxide is a well known multifunctional nanoparticle because of its ubiquity, stability and non toxicity [1]. TiO_2 is used in pigments, optical filters, antireflection coatings, sensors and catalysts [2]. This wide range of uses of TiO_2 is due to its unique electronic and structural properties. TiO_2 exists in three main crystallographic forms; anatase, rutile and brookite. Anatase and rutile forms are of commercial importance, since they are semiconductors with a high energy band gap (3.00 eV for rutile and 3.23 eV for anatase [3]).

TiO_2 can be greatly improved by doping with metal ions, such as nickel, chromium, iron, vanadium, and zinc etc. [4]. Doping opens up the possibility of changing the electronic structure of TiO_2 nanoparticles, altering their chemical composition and optical properties.

Iron has been considered an appropriate candidate, owing to the radius of Fe^{3+} (0.64 Å) being similar to that of Ti^{4+} (0.68 Å). Therefore, it can be inferred that Fe ions might easily be incorporated with the crystal lattice of TiO_2 [5]. One of the interests of Fe-doped TiO_2 is its potential application in spintronic and magneto-optic devices. The band gap of iron is

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2.6 eV, so, it will reduce the width of the energy gap of TiO_2 and increase the efficiency of absorbing visible light [6].

Fe doped TiO_2 could enhance hydrogen production effectively [7]. Furthermore, Fe doped TiO_2 can be used in spintronic devices [8], which would allow for the creation of systems that would consume less energy, and keep and transfer huge amounts of information, depending on the unique combination of ferromagnetic properties of the iron [9].

The material properties seem to be strictly dependent on the crystal structure, the size of the nanoparticle, and morphology, which are correlated with the TiO_2 method of synthesis [10]. There are several methods of synthesising Fe doped TiO_2: sol–gel [11] hydrothermal [12] wet chemical synthesis [13] thermal hydrolysis [14] and plasma oxidative pyrolysis [15,16]. There are also some combined methods, such as combining the sol–gel method with hydrothermal treatment [17]. The most popular method is the sol–gel method, due to the easy technique, low price, the purity of oxides obtained and the lower synthesis temperatures. There are advantages from economical, as well as qualitative, points of view [18].

In this work, we have prepared Fe doped TiO_2 at the Fe/Ti molar ratio (5%). The post annealing of the samples took place at different temperatures; 400 °C, 600 °C and 800 °C. The advantage of our method of preparation is that while there is control over the concentration of dopant, post annealing can be done at different ambient temperatures, in order to tune the amount of oxygen vacancies in the prepared powder.

The current analysis aims to understand the correlation between optical and electronic properties of TiO_2:Fe nanoparticles. The prepared samples have been characterized by XRD, TEM, XPS, and PL.

2. Experimental procedure

2.1. Synthesis of Fe doped TiO_2 Nano-powders

The Fe doped TiO_2 nano-powder was synthesized by the sol–gel method. The powders were prepared at Ti/Fe molar ratios; 1%, 3%, 5%, 8% and 10%. Here, we present only 5% because this sample exhibits good optical properties. The initial sol gel consisted of Iron (III) Chloride 6-Hydrate, $C_{12}H_{28}O_4Ti$ (Tetraisopropyl orthotitanate), ethanol, citrate acid and $C_5H_8O_2$ (Acetyl acetone). The resulting mixture was stirred and dissolved at 40 °C and then refluxed at $T = 120$ °C for 6 h. The sol was further slowly heated at $T = 80$ °C for 14 h in an open bath, in order to cause the required chemical reactions for the development of polymerization and evaporation of the solvent, until a brown wet gel was obtained. During continuous heating at this temperature, the polymerization between citrate acid, acetyl acetone and complexes was developed, and, finally, turned more viscous, and into a wet gel. Finally, the wet gel was fully dried by direct heating on a hot plate at $T = 150$ °C for 1 h. The resulting product was a black-brown porous gel. The aerogel was ground, resulting in a precursor powder called xerogel, which was post annealed to 400 °C, 600 °C and 800 °C for 1 h (in air) and then cooled down to room temperature.

2.2. Characterization of powders

X-ray Diffraction (XRD) analysis was carried out utilizing a D8 Advance Bruker system using $CuK\alpha$ ($\lambda = 0.154056$ nm) radiation and 2θ varying from 20 °C to 70 °C.

High resolution transmission electron microscopy (HRTEM) micrographs of the prepared nanopowders were recorded using a JEOL 2100F Field Emission Gun Transmission Electron Microscope (FEG TEM) operating at 200 keV. The samples were prepared for HRTEM analysis by dispersing the Fe doped TiO_2 nanopowders onto a holey carbon grid.

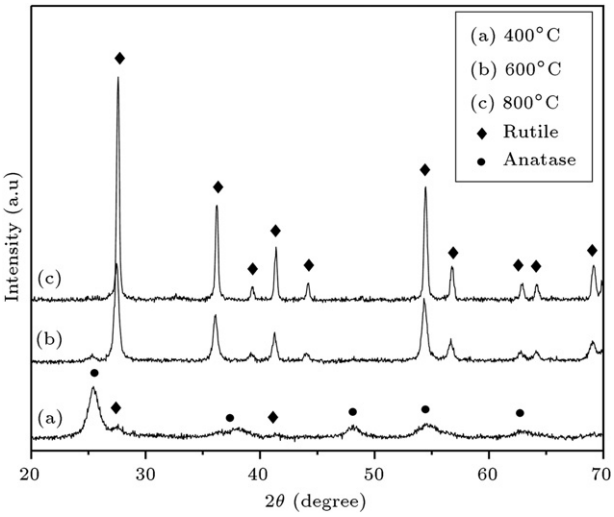

Figure 1: X-ray diffraction patterns of 5% Fe:TiO_2 annealed at (a) 400 °C, (b) 600 °C, and (c) 800 °C.

High-resolution XPS spectra were measured by a Scienta ESCA 300 photoelectron spectrometer in the Daresbury Laboratory at the National Centre for Electron Spectroscopy and Surface Analysis (NCESS), UK. This incorporates a rotating anode, Al $K\alpha$ ($h\upsilon = 1486.6$ eV) X-ray source, a 7 crystal X-ray monochromator and a 300 mm mean radius spherical sector electron energy analyzer with a parallel electron detection system. The X-ray source was run with a 200 mA emission current and 14 kV anode bias while the analyzer operated at 150 eV pass energy with 0.8 mm slits. All measurements were carried out at a pressure in the 10^{-9} Torr range. Sample charging was compensated with an electron flood gun. The binding energy scale of the spectra was referenced to the residual C 1s due to amorphous carbon, which was assigned to a binding energy of 284.6 eV. Collected XPS spectra were fitted using Winspec software.

Luminescence measurements have been performed on the MOLES synchrotron end-station, which has a closed cycled He cryostat [19], using 250 nm diode as the excitation source.

3. Results and discussion

The XRD patterns of prepared Fe doped TiO_2 nano-powders at different doping concentrations and different annealing temperatures, as shown in Figure 1, show that the samples annealed at $T = 400$ °C have both anatase and rutile structures with a dominance of anatase structure. As the annealing temperature is risen to 600 °C, the rutile phase becomes dominant over the anatase phase. Finally, at $T = 800$ °C, the anatase structure disappears and the samples show monostructure.

This pattern is repeated for all different concentrations of Fe in TiO_2. The samples did not show any diffraction peaks of iron or iron compounds, which suggest the formation of an iron-titanium solid solution, where the iron ion has been incorporated into the TiO_2 crystal structure substitutionally, due to similar ionic radii (Ti (0.68 Å) and Fe (0.64 Å)).

It can be seen that a major effect of iron doping seems to be a decrease of the anatase-TiO_2 crystalline phase with the increase of iron concentration and, consequently, an increase

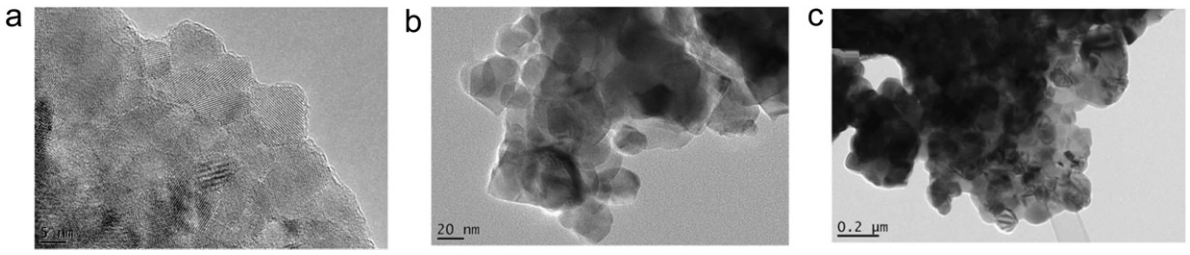

Figure 2: TEM image of 5%Fe:TiO_2 annealed at (a) 400 °C, (b) 600 °C, and (c) 800 °C.

Table 1: Experimental particle size of 5%Fe:TiO_2 annealed at 400, 600, and 800 °C.

	Particle size (nm)	Phase
5%Fe, 400 °C	6–10	Anatase
5%Fe, 600 °C	22–30	Anatase Rutile
5%Fe, 800 °C	50–100	Rutile

in the rutile phase. The TEM images of the 5% Fe doped TiO_2 at $T = 400, 600$, and 800 °C are shown in Figure 2, and the experimental size of the 5% Fe doped TiO_2 at $T = 400$ °C, 600 °C and 800 °C nanoparticles were summarized in Table 1. From Table 1, it can be seen that the samples annealed at 400 °C are in an anatase phase with particle size ranging from 6 to 11 nm. The appearance of the rutile starts at 600 °C (with particle diameter of 22–30 nm) and becomes the dominant phase at 800 °C (with particle diameter of 50–100 nm). It has been shown by Li et al. [20] that anatase to rutile phase transformation only occurs in temperature ranges from 973 to 1073 K, and the particle size of both anatase and rutile phases increases as the temperature increases. But, the growth rate is different, with the rutile phase having a much higher growth rate than the anatase phase. The anatase type to rutile type transformation after doping by iron is accelerated and shifts the transformation to lower temperatures [14], which confirms our results, in which the transformation of anatase to rutile phase has occurred at 600 °C by doping TiO_2 with 5% of Fe. This is evidence that the iron impurity has accelerated the anatase to rutile (A–R) phase transition. In order to analyse the chemical composition of the prepared Fe–TiO_2 nanoparticles, to detect the presence of iron in our samples and identify their chemical state, the samples were characterized by XPS.

Figure 3 shows the high resolution XPS spectra of the Fe 2p region taken on the surface of the 5% Fe-doped TiO_2 nanoparticles prepared at different calcination temperatures ranging from 400 °C to 800 °C. It was found from the binding energies that all Fe-doped TiO_2 samples are richer in Fe^{3+} with a positive shift in binding energies, as shown in Figure 4 and Table 2. By considering the initial sol gel of Iron (III) chloride 6-hydrate, it would be expected that Fe would exist mainly in the +3 oxidative state (Fe^{3+}) [14]. Binding energies in the literature for Fe^{3+} and Fe^{2+} are 710.9 eV and 709.4 eV, respectively [21]. However, the peaks denoted with F^{2+} and surface, in Table 2, could also be due to the main crystal-field split lines of Fe^{3+}[22,23].

The photoluminescence spectrum of 5%: TiO_2 calcined at 400 °C shows an emission peak at around 438 nm (2.83 eV). This active band could be due to the bound excitons [24]. As shown in Figure 5, by increasing the temperature, the photoluminescence intensity decreases, due to nonradiative transition, and peak positions of the photoluminescence are shifted towards the longer wavelength.

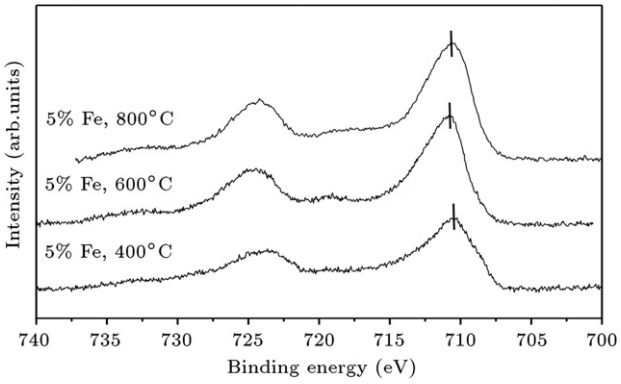

Figure 3: High resolution XPS core level spectra of the Fe2p region of 5% Fe-doped TiO_2 calcined at 400, 600, and 800 °C.

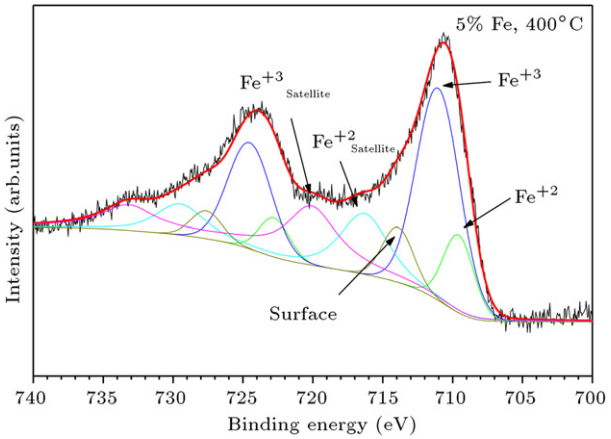

Figure 4: High resolution XPS core level spectrum of 5%Fe calcined at 400 °C.

Table 2: Summary of iron peak positions for 5% Fe calcined at 400 °C.

	2P3/2 (FWHM)	Satellite (FWHM)	Oxidation state
5%Fe_400 °C	709.57 (2.50)	716.30 (4.0)	Fe^{+2} or crystal-field split lines of Fe^{+3}
	711.30 (3.62)	720.10 (4.5)	Fe^{+3}
	714.00 (2.57)	–	'Surface component' or crystal-field split lines of Fe^{+3}

At high concentrations of 10% Fe doped TiO_2, no luminescence could be observed, which can be explained as follows: in low iron concentrations, the iron ions are randomly distributed in the host lattice and the ion-ion distance is too far apart. But, in cases of high concentration, the distances between the ions are shortened, which results in an energy transfer between nearby

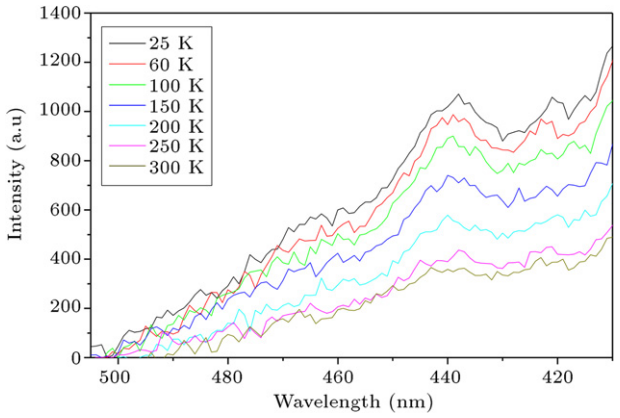

Figure 5: Photoluminescence spectra of 5%Fe:TiO_2 calcined at 400 °C.

ions, so, the concentration quenching process will be the predominant nonradiative decay process at higher concentrations.

4. Conclusions

TiO_2 nanoparticles doped with different concentrations of Fe ions were synthesized by a sol–gel method. The presence of Fe was detected in all samples, in spite of low doping levels, by XPS. The results of the XPS study suggest that Fe ions are predominantly in Fe^{3+} states.

Curve fitting for the various chemical states of Fe containing samples has been presented. These more detailed fitting procedures can be used to resolve and quantify various chemical states in samples containing iron. The sample at 5%Fe calcined at 400 °C shows luminescence, but, by increasing doping concentration, the nonradiative decay process becomes dominant, which is attributed to the concentration quenching process.

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